

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

George K. Burgess, Director

TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS, No. 250

[Part of Vol. 18.]

PULP AND PAPER FIBER COMPOSITION STANDARDS

**REFERENCE STANDARDS, SHOWING THE COLOR
REACTIONS OF COMMON PAPER-MAKING FIBERS
AND STANDARD FIBER MIXTURES WITH VARIOUS
STAINS FOR USE IN IDENTIFICATION AND
ESTIMATION OF FIBER COMPOSITION OF PAPER**

BY

MURIEL F. MERRITT, Laboratory Assistant
Bureau of Standards

April 25, 1924



PRICE, 15 CENTS

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By Muriel F. Merritt.

ABSTRACT.

In standardizing the microanalysis of paper fibers there are no colored paper fiber charts or plates of standard pulps. The need for such plates is felt by beginners and laboratory workers in microanalysis, as the advantage of the colored micrograph over the ordinary photomicrograph is that the former more correctly represents the fibers as observed, since it appeals to one's color sense. Standard pulps and colored plates of standard fiber mixtures greatly aid in the identification of fibers by color and form, and the determination of percentage composition can then be made more easily. This publication covers eight fiber compositions, illustrating pure pulps and mixtures, with their color reactions. These fibers are rag, sulphite, soda, sulphate, ground wood, jute, manila, and esparto. Standard pulps and pulp compositions used by the Bureau of Standards for comparison work in the estimation of fiber content were selected for the micrographs, several slides being prepared for each drawing. Four stains that proved satisfactory in accentuating the details of the fibers were Delafield's hematoxylin (1 per cent solution), malachite green, Herzberg stain, and the Lofton-Merritt stain (malachite green-fuchsine stain) for unbleached sulphite and sulphate. Formulas are given in the text. Magnification of 200 gave the best definition for drawing. The colors of the stained fibers were reproduced by matching the stained fibers with filter paper painted with water colors, the colors used being listed.

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I. INTRODUCTION.

This publication covers eight fiber compositions, illustrating pure pulps and mixtures, with their color reactions. These fibers are rag (linen and cotton), sulphite (coniferous), soda (deciduous), ground wood (coniferous), sulphate (coniferous), jute, manila, and esparto. The fibers selected were those of most importance to the industry.

II. NEED OF MICROGRAPHS.

In standardizing the microanalysis of paper fibers there are no colored fiber charts or plates of 100 per cent pure pulps or standard percentage mixtures of standard pulps. The need for such plates is felt by beginners and laboratory workers in microanalysis. The advantage of the colored micrograph over the ordinary photomicrograph is that the former more correctly represents the fibers as observed, since it appeals to one's color sense. These micrographs

were drawn especially for laboratory needs. Standard pulps and colored plates of standard fiber mixtures greatly aid in the identification of fibers by color and form, and the determination of percentage composition can then be made more easily. (Colored figures 1 to 9 follow last page of text.)

III. METHODS.

Standard pulps and pulp compositions used by the Bureau of Standards for comparison work in estimation of fiber content were selected for the micrographs, several slides being prepared for each drawing. Different stains were used to bring out as many characteristics as possible. For example, in the case of Figure 1 (50 per cent rag and 50 per cent sulphite), one slide stained with Delafield's hematoxylin brought out surface corrugations on both types of fibers, while the Herzberg stain brought out other characteristics. Malachite green was useful in making microscope slides of esparto, because it stained the cells peculiar to that grass a delicate green, whereas the Herzberg stain colored them a deep blue.

Four formulas of stains particularly useful in staining fibers are Delafield's hematoxylin,¹ malachite-green solution, Herzberg stain, and the malachite green-fuchsine stain (Lofton-Merritt stain).

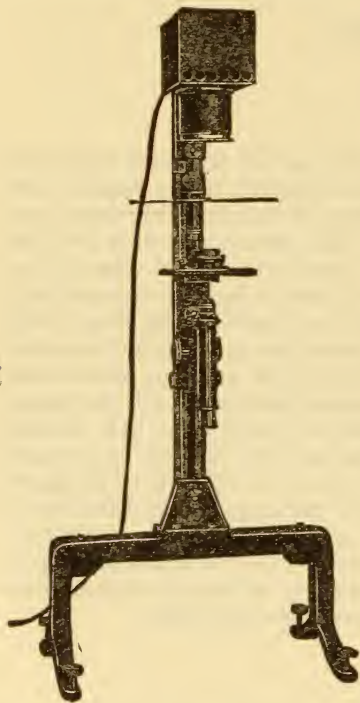


FIG. 10.—Micrographic apparatus.

This is used to project on a sheet of paper the magnified image of paper fibers, for the purpose of tracing the outlines and detailed structure of the fibers.

¹ The Microtommists Vade Mecum, Arthur Bolles Lee, p. 189, Blakiston, Philadelphia, Pa., 1905; Methods in Plant Histology, Charles J. Chamberlin, p. 145, University Chicago Press; 1916.

The Delafield's hematoxylin in our stock was made in 1919. The general formula is: To 400 cm³ of saturated solution of ammonia alum add 4 g of hematoxylin crystals dissolved in 25 cm³ of strong alcohol. Leave exposed to the light and air in an unstoppered bottle three or four days. Filter and add 100 cm³ methanol (CH₃OH). Allow the solution to set until the color is sufficiently dark, then filter and keep in a tightly stoppered bottle.

It is well to allow it to ripen for at least two months before using. As this stain is very powerful, use in the proportion of one drop of stain to two drops of water. Allow to remain on the fibers two minutes, then rinse with water.

MALACHITE GREEN (1 per cent solution).—Dissolve 1 g malachite-green crystals in 100 parts hot distilled water. Let the solution settle overnight and filter with a Gooch filter. Use one drop of stain with one of water and let stay on the fibers two minutes, then rinse with water.

HERZBERG STAIN,² SOLUTION A.—Dissolve 50 g dry zinc chloride (fused sticks) in 25 cm³ distilled water measured with a pipette. The specific gravity of the solution should be 1.8 and must be readjusted if not exactly of this strength. Pour 40 cm³ of the solution into a tall cylinder.

SOLUTION B.—Rinse the vessel and instrument used for solution A with distilled water and dissolve 5.25 g potassium iodide and 0.25 iodine in distilled water, using a total of 12.5 cm³ of water. Add solution B to solution A, stir well and keep in a dark place. The following day pipette off the clear portion into a black bottle, leaving 3 or 4 cm³ of the stain above the sediment. Add a leaf of crystal iodine. Where color differentiations are important, the stain should not be used longer than two weeks. Weights and measures must be carefully observed as Herzberg advised in his directions. For micrographs dilute the Herzberg stain one-half on the microscope slide; otherwise it is too dense.

LOFTON-MERRITT STAIN³ (SULPHITE-SULPHATE), SOLUTION A.—Two grams malachite-green crystals, and 100 cm³ distilled water.

SOLUTION B.—One gram fuchsin (basic) crystals, and 100 cm³ distilled water.

One part malachite green is used with two parts of the fuchsin solution. Two drops of the mixed solution are left on the fibers

² Improvements in method of making the Herzberg stain used in fiber analysis. Muriel F. Merritt. *Paper Trade Journal*, Aug. 24, 1922; New York.

³ Method for differentiating and estimating unbleached sulphite and sulphate in paper. R. E. Lofton and M. F. Merritt, B. S. Tech. Papers, No. 189.

HCl

two minutes, then rinsed with four drops of a weak aqueous solution of hydrochloric acid made by adding 1 cm³ concentrated acid (sp. gr. 1.19: ~~cm~~ 37 per cent) to 1 liter of water. The acid solution is allowed to remain on the fibers 10 seconds.

A magnification of 200 gave the best definition for drawing purposes. As reproduced the micrographs are about 50 magnification. Where practical the fields were drawn as they occurred in the slide, care being taken to get as many representative fibers as typical of the pulp or pulp mixtures. For the color medium water colors were employed. In order to correctly reproduce color reactions of the stains and fibers, rag filter paper was cut in narrow strips and painted with the approximate color sought. These painted strips were then matched under the microscope by daylight with the stained fibers. If the color of the paint was not standard, the paints were mixed until the standard color was produced.

Colors used to match the Herzberg stain (yellow-brown):

Japanese water colors—

1. Brilliant yellow.
2. Deep yellow.

English water colors—

1. Burnt umber.
2. Burnt sienna.
3. Olive green.
4. Van Dyke brown.

Colors used to match the Herzberg stain (red):

English water colors—

1. Brown madder.
2. Rose madder.
3. Prussian blue.

Colors used to match Herzberg stain (blue):

English water colors—

1. Prussian blue.
2. Brown madder.
3. Rose madder.

Colors used to match the Lofton-Merritt stain (purple):

English water colors—

Mauve.

Malachite-green solution.

Colors used to match the Lofton-Merritt stain (green-blue):

English water colors—


Prussian blue.

Malachite-green solution.

IV. CONCLUSION.

These micrographs should be of value to beginners in microanalysis of paper fibers and stimulate the desire to use such plates and create a demand for further plates of this kind. Additional plates of the more important standard fiber, compositions may then be published. The selection contains the most common fibers and fiber mixtures, and these will be found most useful to the paper industry in the standardization of microanalysis.

WASHINGTON, October 9, 1923.



100% RAG



100% SULPHITE



50% RAG—50% SULPHITE

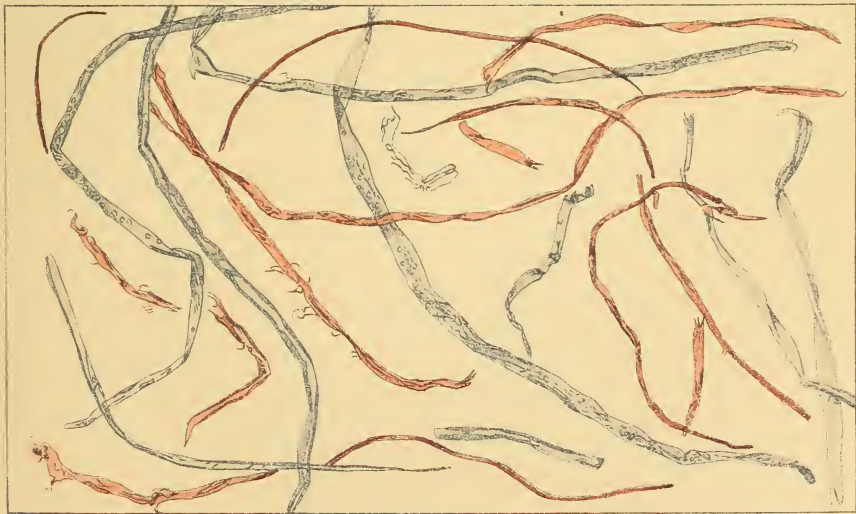
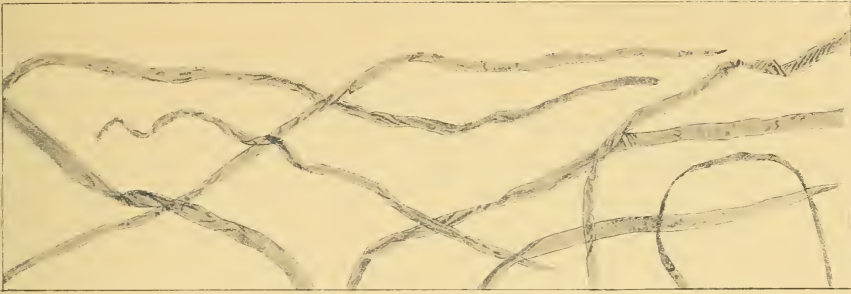


FIG. 1.—Field showing standard-stained fibers of cotton and linen rag pulp and of sulphite (coniferous wood) pulp.

100% SULPHITE



100% SODA



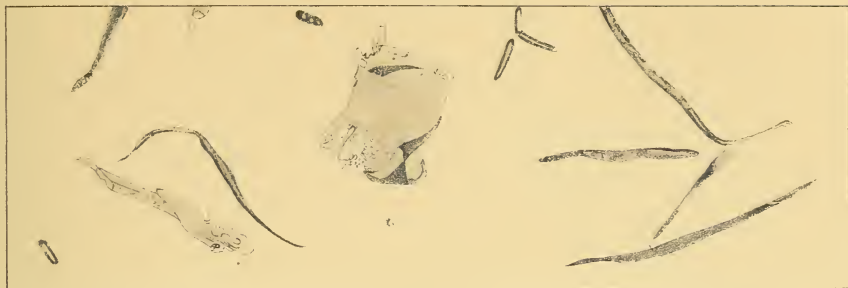
50% SULPHITE—50% SODA



Anscombe & Co. Ltd.

FIG. 2.—Field showing standard-stained sulphite (coniferous wood) and soda, (deciduous wood) pulps.

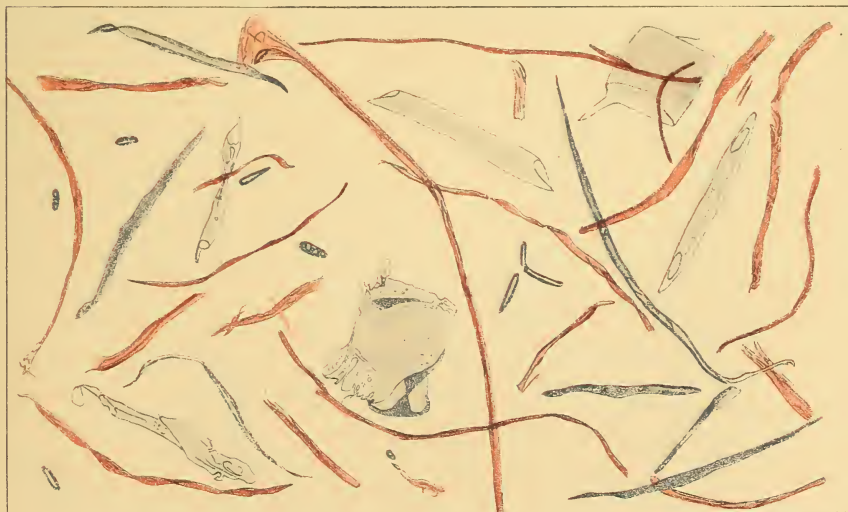
100 % SODA



100 % RAG



50 % SODA—50 % RAG



A. Heen & Co. Baltimore

FIG. 3.—Field showing standard-stained fibers of soda (deciduous wood) and of linen and cotton rag pulp.

100% GROUNDWOOD



100% SULPHITE



50% GROUNDWOOD—50% SULPHITE



FIG. 4.—Field showing standard-stained fibers of groundwood and sulphite fibers (coniferous wood)pulps.

A. Hoen & Co. Baltimore

100% JUTE



100% MANILA



50% JUTE—50% MANILA



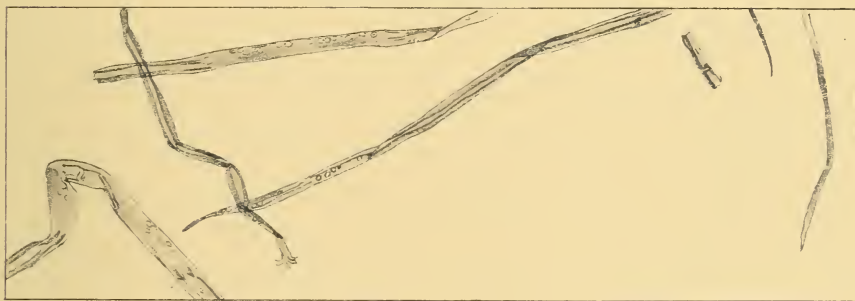
FIG. 5.—Field showing standard-stained fibers of jute and manila pulp.

A. H. H. & Co. Baltimore

50% JUTE—50% MANILA



100% SULPHITE



50% JUTE-MANILA—50% SULPHITE



FIG. 6.—Field showing standard-stained fibers of jute, manila and sulphite coniferous pulps.

A. Hagen & Co. Boston

100% ESPARTO



100% SULPHITE



50% ESPARTO—50% SULPHITE

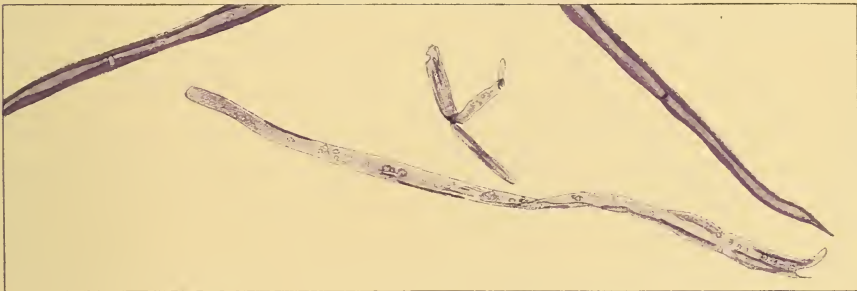


FIG. 7.—Field showing standard-stained fibers of esparto and sulphite (coniferous wood) pulp.
(Reproduced by the author from standard samples of 50% esparto—50% sulphite pulp obtained from England)

100% SULPHATE



100% SULPHITE



50% SULPHATE—50% SULPHITE



FIG. 8.—Field showing standard-stained fibers of sulphate and sulphite (coniferous wood) pulp.

STANDARD COLOR SHADES OF STANDARD STAINS

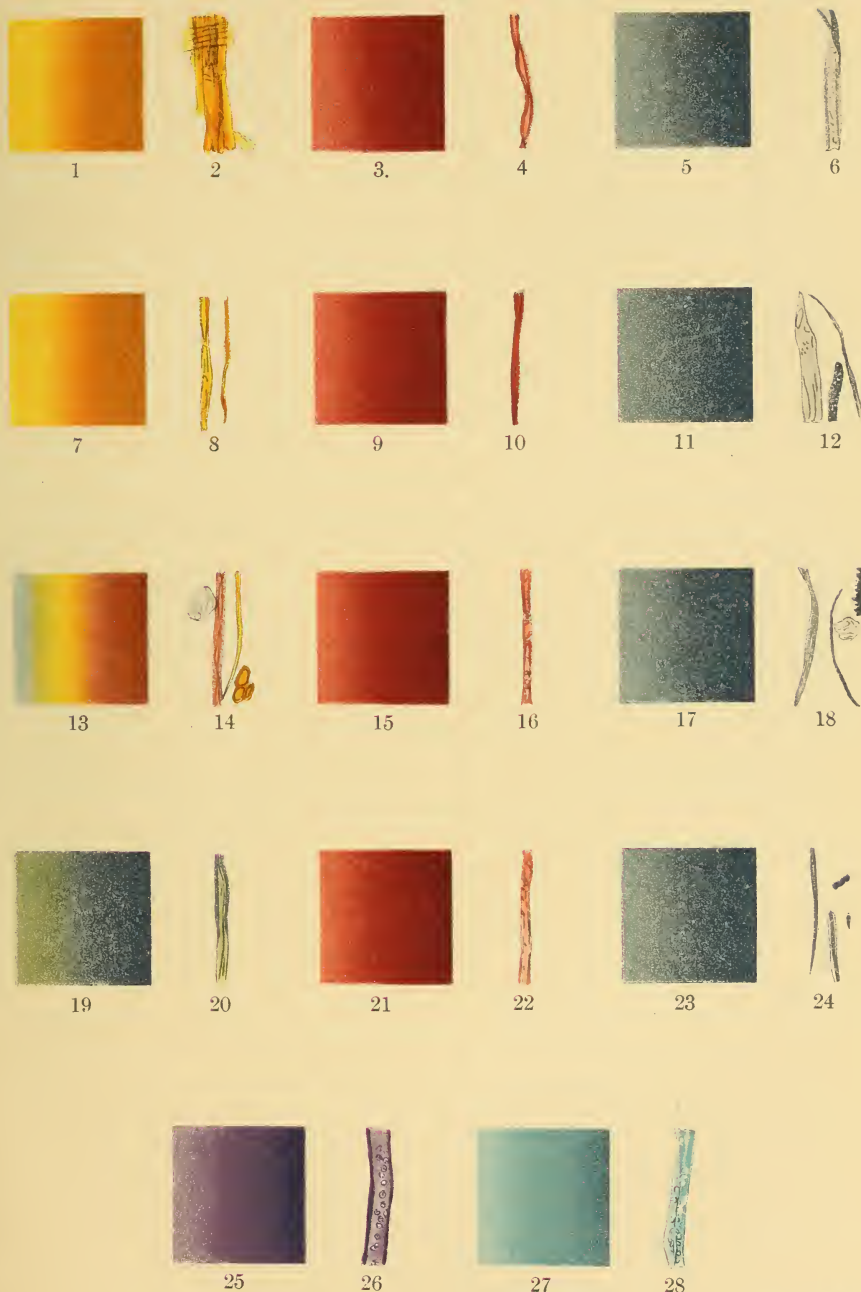


FIG. 9.—Standard shades of standard-stained colors for use in identification and estimation of fiber composition.

Explanation of Figures: No. 2, mechanical or ground wood; 4, cotton; 6, bleached sulphite (coniferous); 8, jute; 10, flax (linen); 12, soda (deciduous); 14, manila (*musa textilis*); 16, ramie; 18, rice straw; 20, unbleached sulphite (coniferous); 22, carua; 24, esparto; 26, unbleached sulphate (coniferous); 28, unbleached sulphate (coniferous).

Herzberg stain No. 1-24. Lofton-Merritt stain No. 25-28.

A. Hoen & Co. Baltimore

